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ABSTRACT

Soil and debris from Superfund sites must be treated to minimize their threat to human health and the environment as part of remedial actions at such sites. Studies were conducted on the effectiveness with which five treatment processes removed or immobilized synthetic soils containing volatile and semivolatile organics and metals. The treatment technologies were: soil washing, dechlorination with potassium polyethylene glycol (KPEG), incineration, low temperature thermal desorption and solidification/fixation. This paper describes the production of four synthetic soils containing varying levels of contaminants and reports the effectiveness of the five treatment methods.

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INTRODUCTION

The RCRA Hazardous and Solid Waste Amendments of 1984 prohibit the continued land disposal of untreated hazardous wastes beyond specified dates. The statute requires the U.S. Environmental Protection Agency (EPA) to set "levels or methods of treatment, if any, which substantially diminish the toxicity of the waste or substantially reduce the likelihood of migration of hazardous constituents from the waste so that short-term and long-term threats to human health and the environment are minimized." The legislation sets forth a series of deadlines beyond which further disposal of untreated wastes is prohibited. Specifically, Sections 3004(d)(3) and (e)(3) require solid/debris waste material resulting from a Superfund-financed response action or an enforcement authority response action implemented under Sections 104 and 106 of CERCLA,** respectively, to become subject to the land ban on November 8, 1988.

In response to this mandate, the EPA Office of Solid Waste and Emergency Response (OSWER) is developing standards for the treatment of these wastes. These standards will establish treatment levels through the evaluation of readily available treatment technologies. In the future, Superfund wastes meeting these levels or standards may be deposited in land disposal units; otherwise, they will be banned from land disposal unless a variance is issued. EPA's Office of Research and Development has initiated a research program to

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** Comprehensive Environmental Response, Compensation, and Liability Act

identify and evaluate readily available treatment technologies for contaminated Superfund soils.

Under Phase I of EPA's research program, which was conducted from April to November 1987, a surrogate soil containing a wide range of chemical contaminants typically occurring at Superfund sites was prepared and subjected to bench- or pilot-scale performance evaluations using the following treatment technologies: 1) physical separation/volume reduction (soil washing), 2) chemical treatment (specifically, KPEG), 3) thermal desorption, 4) incineration, and 5) stabilization/fixation. This report covers the formulation and development of the surrogate soil; it also highlights the results of the five treatment evaluations. It is worth noting that virtually all of the analytical data underlying this research were developed using EPA-SW846 methods. Detailed project reports covering the findings of each study are available through EPA's Hazardous Waste Engineering Research Laboratory in Cincinnati (see acknowledgments for contact names).

PREPARATION OF SURROGATE SOIL (SARM)

SARM, an acronym for synthetic analytical reference matrix, is the term used throughout this text to refer to the synthetic soil. The decision to use a synthetic soil was driven by several factors. First, RCRA permit regulations restricting off-site treatment of hazardous wastes, such as contamination Superfund site soils, limited the planned research program. Second, there was a strong desire for the test soil to be broadly representative of a wide range of soils and contaminants, and it was felt that no single site soil could adequately satisfy this need. Third, large quantities of a homogeneous test material were needed for the research program, particularly for incineration, which was to be evaluated using pilot-scale equipment (requiring thousands of pounds of feed stock). Fourth, it was important to have contaminants present in the soil at sufficient levels to determine at least 99 percent reduction efficiencies. Fifth, the contaminants had to include both metals and organics, and the organics had to include compounds representing a wide variety of structural types (e.g., both chlorinated and nonchlorinated alphatics and aromatics, volatiles and semivolatiles, etc.). Sixth, the soil with its mix of contaminants had to present a reasonable challenge to the technologies of interest.

The basic formula for the SARM soil was determined from an extensive review of 86 Records of Decision (ROD's) and a parallel independent study of the composition of eastern U.S. soils. The recommendations of both sets of data came to almost the same conclusion: 30 percent by volume of clay (montmorillinite and kaolinite), 25 percent silt, 20 percent sand, 20 percent top soil, and 5 percent gravel. These components were assembled, air-dried, and mixed together in two 15,000-lb batches in a standard truck-mounted cement mixer.

Also, as part of the background work, the ROD's were studied to determine the occurrence, frequency, and concentration of more than 1000 contaminants found on Superfund sites. The objective of this effort was to identify contaminant groups, and indicator chemicals for those groups, that were most representative of CERCLA wastes.

The three basic contaminant groups identified as being frequently found in Superfund site soil and debris were volatile organics, semivolatile organics, and metals. The selection of specific compounds to serve as representative analytes for each contaminant group was based on an analysis of specific site contaminants and their occurrence, as well as the physical and chemical properties of each compound, including:

- Molecular structure
- Vapor pressure
- Heat of vaporization
- Heat of combustion
- Solubility
- Henry's Law constant
- Partition coefficient
- Soil adsorption coefficient

Health effects and toxicity were also taken into account during the selection process.

As a result of this research effort, a list of target contaminant compounds was developed that represented the most frequently occurring hazardous compounds at Superfund sites, and that also provided a challenging test matrix for all five treatment technologies. The final list of chemical contaminants chosen for the SARM studies is as follows:

Volatile organics	Metals
Ethylbenzene	Lead
Xylene	Zinc
1,2-Dichloroethane	Cadmium
Tetrachloroethylene	Arsenic
Acetone	Copper
Chlorobenzene	Chromium
Styrene	Nickel

Semivolatile organics

Anthracene
Pentachlorophenol
Bis(2-ethylhexyl)phthalate

The final step in this research process was to examine the levels at which these chemicals have been found at Superfund sites and to select concentrations that would be representative of contaminated soils and debris. The EPA compiled average and maximum concentrations of each selected chemical and calculated the percentage of each compound within its group. From these data, target contaminant concentrations were devised for formulating four different SARM preparations:

SARM 1: High levels of organics (20,800 ppm volatiles plus 10,000 ppm semivolatiles) and low levels of metals (1,000 ppm total metals).

SARM 2: Low levels of organics (2,080 ppm volatiles plus 1,000 ppm semivolatiles) and low levels of metals (1,000 ppm total metals).

SARM 3: Low levels of organics (2,080 ppm volatiles plus 1,000 ppm semivolatiles) and high levels of metals (50,000 ppm total metals).

SARM 4: High levels of organics (20,800 ppm volatiles plus 10,000 ppm semivolatiles) and high levels of metals (50,000 ppm total metals).

Table I presents the selected target levels for each of the contaminants in each of the four SARM's.

More than 28,000 pounds of SARM samples were prepared through a series of small-scale mixing operations utilizing commercial stocks of chemicals, the clean SARM soil, and a 15-ft^3 mortar mixer. Batches of each SARM were prepared in 500-lb quantities sufficient to meet the needs of each treatment technology. Only a few pounds of each SARM was necessary for most of the technologies because they were conducted at bench scale; however, incineration was evaluated at pilot scale, and therefore required thousands of pounds of SARM to serve as feed stock for the testing. More than 200 lb of each SARM was also reserved, packaged, and archived for future use. The archived samples are currently being stored at EPA's R&D facility in Edison, New Jersey, and are available to serve as standard test material for future treatability studies.

A number of chemical and physical analyses of the basic SARM soil and the four spiked SARM formulas have been conducted to verify their composition prior to treatability testing. Results of the physical and chemical analyses are compiled in Tables II through IV. Toxicity characteristic leaching procedure (TCLP) data were also generated during the study, but space limitations prevent their being presented here. These data can be found in the individual EPA project reports.

METHODOLOGY AND RESULTS OF TREATMENT EVALUATIONS

Physical Separation/Volume Reduction (Soil Washing)

As part of the performance evaluation of this technology, samples of each SARM were physically treated in a series of bench-scale washing experiments designed to simulate the EPA-developed pilot-scale Mobile Soils Washing System (MSWS). This system physically separates contaminated fines from coarse soil material, which effectively reduces the volume of the contaminated portion of the soils. The MSWS is expected to be an economic alternative to the current practice of hauling contaminated soils offsite to a landfill and replacing the excavated volume with fresh soils. The use of a soil washing system also performs the task of feedstock preparation for other subsequent treatment technologies by prescreening the soil into a "smooth" homogenous feed.

Specifically, this project was designed to simulate the drum-screen washer segment of the MSWS. This segment separates the $\geq$2-mm soil fraction (coarse material) from the $\leq$2-mm soil fraction (fines) by use of a rotary drum screen. A high-pressure water knife operates at the head of the system

TABLE I. TARGET CONTAMINANT CONCENTRATIONS FOR SARMS
(mg/kg)

Analyte	SARM I (High organic, low metal)	SARM II (Low organic, low metal)	SARM III (Low organic, high metal)	SARM IV (High organic, high metal)
Volatiles				
Acetone	6,800	680	680	6,800
Chlorobenzene	400	40	40	400
1,2-Dichloroethane	600	60	60	600
Ethylbenzene	3,200	320	320	3,200
Styrene	1,000	100	100	1,000
Tetrachloroethylene	600	60	60	600
Xylene	8,200	820	820	8,200
Semivolatiles				
Anthracene	6,500	650	650	6,500
Bis(2-ethylhexyl) phthalate	2,500	250	250	2,500
Pentachlorophenol	1,000	100	100	1,000
Inorganics				
Arsenic	10	10	500	500
Cadmium	20	20	1,000	1,000
Chromium	30	30	1,500	1,500
Copper	190	190	9,500	9,500
Lead	280	280	14,000	14,000
Nickel	20	20	1,000	1,000
Zinc	450	450	22,500	22,500

TABLE II. Results of Clean Soil Matrix Analyses.[a]

Sample	Sample and Batch Numbers										
	1	2	3	4	5	6	7	8	9	10	
Batch	1	2	2	1	1	2	2	1	1	2	Average
Cation exchange capacity, meq 100/g	117.5	152.5	150	150	77.5	150	155	80	147.5	147.5	133
TOC, %	3.2	3.9	3.0	3.8	2.8	2.7	-[b]	-	-	-	3.2
pH, S.U.	8.0	9.0	8.5	8.5	9.0	8.0					8.5
Grain size distribution, %											
Gravel	3	2	4	3	2	3	-	-	-	-	3
Sand	55	57	58	54	56	57	-	-	-	-	56
Silt	29	30	27	30	28	27	-	-	-	-	28
Clay	13	11	11	13	14	13	-	-	-	-	12

[a] The clean SARM was also analyzed for all contaminants on the Hazardous Substances List to determine background contamination, if any. Organic analyses showed no volatile or semivolatile compounds at the micrograms/kilogram level; metals analyses showed appreciable quantities of iron, potassium, aluminum, calcium, and magnesium (as would be expected), but no substantial amounts of the more toxic metals (e.g., chrome, nickel, lead, zinc). In other words, the clean SARM was found to be free of anthropogenic contamination.

[b] A dash indicates that the sample was not analyzed for this parameter.

TABLE III. MOISTURE CONTENT OF SPIKED SARMS[a]
(percentage)

Laboratory	SARM-I	SARM-II	SARM-III	SARM-IV	Method
IT Corp. (thermal desorption for PEI)	16.9	6.0[b]	--	--	Oven-dried
Hittman-Ebasco (stabilization for Acurex)	31.4	8.6[b]	19.3	22.1	Oven-dried
Radian Corp. (incineration for PEI)	17.1 16.1 16.1	16.0[c] 17.8[c] 17.6[c]	-- -- --	-- -- --	Oven-dried Oven-dried Oven-dried
EPA - Edison (soil washing for PEI)	22.9 19.6	7.2[b] 6.2[b]	20.6 18.6	30.1 --	Oven-dried Dean Stark distillation
Analytical Enterprises (KPEG for Wright State)	--	--	--	--	
Average (all values)	20.0	11.3 7.0[b] 17.1[c]	19.5	26.1	

[a] Values obtained by the oven-drying method (ASTM D2216) are expressed as percent total moisture (i.e., water plus volatile organics); values obtained by Dean Stark distillation Method (ASTM D95) represent percent water only.

[b] These values are for aliquots taken only from Batch 1 of SARM-II, to which only a small amount of water was added. See footnote C.

[c] These values are for subsequent batches of SARM-II, which were prepared with a higher water content, similar to that added to other SARMs.

TABLE IV. ANALYTICAL PROFILE OF SPIKED SARM'S: AVERAGE CONCENTRATIONS FOUND UPON TOTAL WASTE ANALYSIS[a] (mg/kg)

Analyte	SARM I (High organic, low metal)	SARM II (Low organic, low metal)	SARM III (Low organic, high metal)	SARM IV (High organic, high metal)
Volatiles				
Acetone	4,353 (9)	356 (8)	358 (2)	8,030 (2)
Chlorobenzene	316 (9)	13 (6)	11 (2)	330 (2)
1,2-Dichloroethane	354 (9)	7 (8)	5 (2)	490 (2)
Ethylbenzene	3,329 (9)	123 (8)	144 (2)	2,708 (2)
Styrene	707 (9)	42 (8)	32 (2)	630 (2)
Tetrachloroethylene	408 (9)	19 (8)	20 (2)	902 (2)
Xylene	5,555 (9)	210 (8)	325 (2)	5,576 (2)
Semivolatiles				
Anthracene	5,361 (9)	353 (7)	181 (3)	1,920 (3)
Bis(2-ethylhexyl) phthalate	1,958 (9)	117 (7)	114 (3)	646 (3)
Pentachlorophenol	254 (9)	22 (7)	30 (3)	80 (3)
Inorganics				
Arsenic	18 (10)	17 (7)	652 (4)	500 (4)
Cadmium	22 (8)	29 (6)	2,260 (2)	3,631 (2)
Chromium	24 (8)	28 (6)	1,207 (4)	1,314 (4)
Copper	231 (10)	257 (8)	9,082 (4)	10,503 (4)
Lead	236 (10)	303 (8)	14,318 (4)	14,748 (4)
Nickel	32 (10)	38 (8)	1,489 (4)	1,479 (4)
Zinc	484 (8)	642 (6)	31,871 (4)	27,060 (4)
Moisture, %	20 (7)	11 (7)	19 (3)	26 (2)

[a] Values in parentheses indicate number of samples analyzed.

to break up soil lumps and strip the contaminants off the soil particles. Both the design of the pilot-scale MSWS and the design of the bench-scale experiments to simulate the MSWS for cleanup of the SARMS samples are based on a set of assumptions that underlie the volume-reduction approach of treating contaminated soil, i.e.:

1) A significant fraction of the contaminants are either physically or chemically bound to the silt, humus, and clay particles.

2) The silt and clay are attached to the sand and gravel by physical processes (primarily compaction/adhesion).

3) Physical washing of the sand/gravel/rock fraction will effectively remove the fine sand, silt, and clay-sized (less than 0.2 mm) materials from the coarse material.

4) The contaminants will be removed to the same extent that the silt and clay are separated from the sand/gravel/rock fraction (i.e., increasing the efficiency of the washing process will directly increase the removal efficiency for the majority of the contaminant mix).

These assumptions were tested by evaluating different wash solutions in a series of bench-scale shaker-table experiments. Two wash solutions were chosen for evaluation: 1) a chelant solution (tetrasodium salt of EDTA, Dow Chemical Versene 100®), and 2) an anionic surfactant solution (phosphated formulation from Procter & Gamble, Institutional Formula Tide®). Organic solvents and oxidizing agents were considered, but were found unacceptable because of material-handling problems associated with these compounds, especially when used in a field situation. Following shaker-table washing, each SARM soil was wet-sieved to separate the fines from the coarse material. Although the EPA MSWS only separates the soil into ≥2-mm and <2-mm size fractions, three size fractions (≥2-mm, 250-μm to 2-mm, and <250-μm) were investigated in this study to determine if the middle fraction (medium to fine sand) could be cleaned effectively and thereby increase the potential volume reduction. For determination of the effectiveness of the soil-washing techniques in reducing the volume of contaminated material, each individual treated size fraction was analyzed for residual total organics and metals by standard gas chromatography/mass spectrometry (GC/MS) and inductively coupled plasma (ICP) techniques (SW-846, 3rd ed.), and for leachable constituents by toxicity characteristic leaching procedures (TCLP, Federal Register Vol. 51, No. 114, June 13, 1986).

The soil-washing experiments were conducted in two phases. During the initial phase, pH and temperature variations were evaluated as well as different wash concentrations of chelant and surfactant. Experiments were also run to determine the optimum reaction time for both the chelant and surfactant solutions. Temperature ranges from 78° to 120°F had little effect on the contaminant reduction efficiencies. The pH of the surfactant solution was adjusted from 5.0 to 12.0 with no appreciable change in the organic contaminant removal efficiencies. A reduction of the pH of the chelant solution to 8.0 produced no additional metal removal (ambient pH of the chelant solution was 12.0).

The optimum chelant concentration was determined to be a 3:1 molar ratio of tetrasodium EDTA to total contaminant metals present in the SARM. A surfactant solution of 0.5 percent (by weight) proved to be most effective in removing the organic contaminants. Reaction times of 15 minutes for the chelant solution and 30 minutes for the surfactant solution were determined to be optimum for allowing sufficient contact between the solution and soil matrix.

During the second phase of these experiments, the optimum conditions for reducing organic and metal contamination (as determined in the initial phase of the soil experiments and discussed in the preceding paragraphs) were applied to all four SARM's and compared with a baseline tap-water wash for each SARM. Tables V through VII show an approximation of the effectiveness of various treatment solutions (wash solutions) by presenting the overall removal efficiencies observed for each size fraction and contaminant group. These efficiencies, which are expressed as percentage reductions, were developed by dividing the residual contaminant concentration in each size fraction by the initial concentration in the whole soil. Although this comparison is admittedly imprecise, it is nevertheless useful for demonstrating trends and relationships between soil fractions, contaminant types, and waste solutions. The discussion that follows examines the data according to the results achieved for each soil size fraction.

The data underlying Tables V through VII clearly showed the tendency for contaminants to accumulate or concentrate in the smaller size fractions (i.e., to bind to the clay and silt). For nearly all of the contaminants, the concentration increased as the size fraction decreased. This finding is consistent with the findings of earlier soil-washing tests.[1,2,3]

For the $\geq$2-mm soil fraction (see Table V), the water wash, the 3:1 molar chelant wash, and 0.5 percent surfactant wash were all about equally effective. In all cases, overall contaminant removal efficiencies by group exceeded 90 percent, and volatile removals as a whole exceeded 99 percent across the board. Semivolatile removals ranged from 90 to 99+ percent, and metals from 92 to 98 percent. Individual contaminant removal efficiencies within groups varied somewhat. These variations are probably due to physical properties associated with each contaminant (such as water solubility, volatility, polarity, etc.), as well as physical properties of the soil (e.g., cation exchange capacity, surface area) and the wash solution itself (pH, temperature, chelant, surfactant concentration, contact time, etc.). These excellent results are believed to be closely related to the "freshness" of the soil. It has been hypothesized that the physical processes of compaction and adhesion were not highly operative in the SARM soils, which allowed the loosely attached silt and clay particles to be easily separated from the larger sand and gravel fractions. These physical attractions tend to be more operative in older soils, and are especially noticeable in soils that have experienced long periods of weathering and contact time between contaminants and soil particles. Because the SARM was a freshly prepared synthetic mixture, the forces of compaction and adhesion at the time of treatment were probably weak, a condition more typical of a recent spill-site soil than an older soil found at an abandoned CERCLA site. Consequently, in these studies, the water

Table V. Soil Washing Effectiveness (greater than 2-mm size fraction),[a] overall percentage reduction by contaminant group.

	SARM I (high organics, low metals)		SARM II (low organics, low metals)			SARM III (low organics, high metals)		SARM IV (high organics, high metals)		
	Water	Surfactant	Water	Surfactant	Chelant	Water	Chelant	Water	Surfactant	Chelant
Volatiles	>99.9	>99.8	99.9	99.9	>99.9	>99.9	99.9	>99.9	99.9	>99.9
Semivolatiles	98.9	>99.8	93.9	93.5	90.1	>94.8	96.4	97.8	>98.3	97.8
Inorganics	92.2	91.5	>96.7	95.7	95.9	98.0	98.4	97.1	98.4	98.1

[a] Total waste analysis.

Table VI. Soil Washing Effectiveness (250-μm to 2-mm size fraction),[a] overall percentage reduction by contaminant group.

	SARM I		SARM II			SARM III		SARM IV		
	Water	Surfactant	Water	Surfactant	Chelant	Water	Chelant	Water	Surfactant	Chelant
Volatiles	99.8	99.8	>99.9	>99.8	>99.9	>99.3	99.0	>99.7	>99.7	>99.7
Semivolatiles	56.2	65.6	52.7	47.3	67.5	0	0	0	29.4	32.3
Metals	81.6	80.7	>82.7	91.6	85.1	96.4	98.4	90.7	91.8	90.3

[a] Total waste analysis.

Table VII. Soil Washing Effectiveness (less than 250-μm size fraction),[a] overall percentage reduction by contaminant group.

	SARM I		SARM II			SARM III		SARM IV		
	Water	Surfactant	Water	Surfactant	Chelant	Water	Chelant	Water	Surfactant	Chelant
Volatiles	66.2	88.0	>99.8	>99.4	99.6	86.7	>93.2	>69.6	95.0	81.8
Semivolatiles	59.7	43.2	0	0	0	0	0	0	0	0
Metals	0	0	0	0	21.9	0	78.2	0	7.3	82.2

[a] Total waste analysis.

wash proved to be as effective in cleaning the ≥2-mm soil fraction as the water-plus-additive solutions.

Contaminant removals from the 250-μm to 2-mm size fraction are summarized in Table VI. Overall, the data show that the volatiles also were efficiently removed from this soil category at levels exceeding 99 percent by all wash solutions. These results are similar to those seen in the ≥2-mm fraction. Semivolatile removal efficiencies dropped off compared with results for the ≥2-mm size fraction (see Table V). Also, semivolatile removal efficiencies for SARM's III and IV were markedly lower than for SARM's I and II. Metal removal efficiencies were also somewhat lower across the board for this size fraction compared with the ≥2-mm fraction. The trend toward reduced removal efficiencies for the semivolatiles and metals is not surprising, as this size fraction has more surface area than the ≥2-mm fraction, and also some small amount of silt and clay particles; therefore, it has a higher potential to adsorb and retain more contamination than the larger ≥2-mm fraction.

For the fine soil fraction (<250 μm) washing with any of the solutions effectively removed the volatiles; conversely, none of the solutions were found to be consistently effective in removing the semivolatiles from this size fraction of the SARM's. Removal of metallic contaminants definitely appeared to be enhanced somewhat by the use of the chelant. As shown in Table VII, the chelant wash was much more effective than with the water wash or the surfactant wash in reducing metal contamination in the fine soil fraction.

In summary, the results support the basic assumptions underlying the volume-reduction approach to soil decontamination; that is, a significant fraction of the contaminants are attached to the fines (silt, humus, and clay), and the coarse material (sand and gravel) can be cleaned by physical separation from the fines. The data indicate that 1) water alone can efficiently remove a significant portion of both the organic and inorganic contamination from the ≥2-mm soil fraction, and 2) the addition of a chelant can enhance metals removals from the middle (2 mm to 250 μm) and fine (<250 μm) soil fractions.

Chemical Dechlorination/KPEG

Chemical dechlorination was examined as a treatment technology because it had already been successfully demonstrated at laboratory scale with PCB- and dioxin-contaminated soils and sludges, and was viewed as a promising treatment technology for development to pilot scale and possibly full scale. The KPEG dechlorination process involves the application of a potassium hydroxide-polyethylene glycol reagent to contaminated soil at elevated temperatures for a period of 2 to 4 hours, after which the reagent is decanted and recovered and the soil is rinsed and neutralized. The reagent strips one or more chlorine atoms from the PCB or dioxin molecule, forming an inorganic chloride salt and a derivative of the PCB or dioxin, which, in theory, should be less toxic than the original contaminant.

Each of the four SARM's was evaluated in this study. Although the SARM's did not contain any PCB's or dioxins, other chlorinated species were present, and there was interest in learning whether these compounds could be dechlorinated. There was also interest in learning whether the process would exhibit any removal effectiveness on the other organic and inorganic contaminants in the test soils.

Testing was conducted in either 500-ml or 2-liter glass reaction vessels mounted within temperature-controlled heating mantles. In each test, either 125 or 500 g of SARM were treated with KPEG reagent at 100°C for 2 hours. During the reaction period, the contents of the glass reaction vessel were continually stirred at 100 rpm with a Teflon-coated stainless steel stirring rod. The system was also continually purged with nitrogen, and the off-gases were filtered through a Tenax/XAD-2/carbon trap system. The contents of the traps were subsequently analyzed to establish material balances and to determine which compounds had been destroyed versus those which had simply been volatilized. At the end of the 2-hour reaction period, the reagent was separated from the soil by centrifugation and decantation. The soil was then neutralized by an acid rinse followed by a plain water rinse. All rinse solutions, soil residues, and the spent reagent were analyzed for the target SARM contaminants.

Overall results of the KPEG tests are given in Table VIII. The analyses show that the KPEG process was very effective in removing the volatiles from all four SARM's. Removal rates for all volatiles exceeded 90 percent in all tests, and most often ranged from 98 to 99+ percent. Although material balances were generally poor, the data strongly indicated that most of the volatiles were unaffected chemically by the treatment and were removed strictly

Table VIII. KPEG effectiveness on SARM's - overall percentage reduction by contaminant group.[a]

	SARM I		SARM II		SARM III		SARM IV	
	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2
Volatiles (all)	99.9	98.3	98.2	96.3	99.5	97.5	99.9	98.1
Semivolatiles								
Anthracene	91.3	96.3	75.6	-10	-490	-1246	96.0	97.0
Pentachlorophenol	98.1	97.7	91.9	94.5	99.6	99.0	95.8	95.4
Inorganics (all)	44.5	-	39.4	-	49.4	-	29.3	-

[a] As measured by total waste analysis. A negative percent reduction results when chemical analysis of a treated residue yields a higher contaminant concentration than the untreated material.

by volatilization processes. Notable exceptions to this were 1,2-dichloroethane and tetrachloroethylene, which appeared to have been completely destroyed by the process.

Semivolatile results are available for only anthracene and pentachlorophenol. In the case of pentachlorophenol, the data indicate it was removed from the soil at efficiency levels ranging from 92 to 99 percent; however, the mass balance data indicate that it was not dechlorinated by the KPEG reagent. Anthracene also was not destroyed. Removal efficiency data for the compound are somewhat equivocal; in the tests utilizing SARM's I and IV, which had starting concentrations of anthracene of 4000+ ppm, it was found to be efficiently removed (i.e., removal rates ranged from 91 to 97 percent). In tests involving SARM's II and III, which had much lower anthracene levels (i.e., less than 250 ppm), no removal was observed. This may be due to analytical limitations associated with recovering anthracene at these levels in soils.

The KPEG process had only a limited effect on removing the inorganic contaminants from the SARM's. Overall removal rates ranged from 29 to 49 percent.

Low-Temperature Thermal Desorption

The purpose of this research was to investigate the capability of a laboratory-scale low-temperature thermal desorption technology for removing volatile and semivolatile contaminants from the SARM's. The laboratory testing program consisted of 15 separate bench-scale tests (10 in a tray furnace and 5 in a tube furnace). Only SARM's I and II were tested at 150°, 350°, and 550°F for 30 minutes to determine the effect of each temperature on removal of the contaminants. The tray furnace was used as a baseline technology to determine the overall effectiveness of thermal desorption in removing contaminants from the soil. The tube furnace was used to provide additional data on the concentration of contaminants in the off-gas in an attempt to establish a material balance.

The first series of 10 tests involved the use of the tray furnace in which SARM's I and II were each tested once at 150° and 350°F (four tests) and three times each at 550°F (six tests). The second series of five tests involved the use of the tube furnace to evaluate the nature of the off-gas (desorbed volatiles) generated during thermal treatment. One tube furnace test was run at 150° and 350°F and three tube furnace tests were run at 550°F using only SARM I. For the tray furnace, the bed of soil that was heated represented the entire sample that was analyzed. For the tube furnace, all of the off-gas was collected as one sample, and the remaining soil residue was collected as a second sample.

Table IX shows the overall results for the tray tests for SARM's I and II. The studies showed that volatiles were efficiently removed from the soil by at least 95 percent at all temperatures. Semivolatiles were removed less efficiently than the volatiles at 150° and 350°F, but removals tended to increase with temperature and approached the 90 percent efficiency range when

550°F was applied. The apparent increase in metal concentrations in the residues (as indicated in the negative reduction values) may be an artifact in the data, due to moisture losses during heating; because the SARM's contained 6 to 17 percent moisture before treatment (see Table III), the losses tended to produce a higher metal-to-soil ratio (i.e., concentration) in the treated residual, which results in an apparent (but unreal) increase in metal content. A second factor that may have contributed to the change in concentration of the metals may have been a change in the matrix's ability to retain metals after heating.

Table IX. Low temperature desorption - overall percent reduction of contaminants by group at various test temperatures using tray furnace and 30-minute residence time.[a]

	SARM I			SARM II		
	150°F	350°F	550°F	150°F	350°F	550°F
Volatiles	97.8	99.8	99.8	98.3	95.9	96.0
Semivolatiles	-5.3	41.6	93.6	11.7	74.8	86.3
Metals	-9.3	-12.1	-15.1	5.1	10.2	-7.3

[a] As measured by total waste analysis. A negative percent reduction results when chemical analysis of a treated residue yields a higher contaminant concentration than the untreated material.

In terms of total actual residual concentrations, the following statements can be made (refer to Table IV for initial concentrations prior to treatment):

SARM I:

- At 350° and 550°F, all volatiles except acetone were reduced to less than 1 mg/kg in the treated residue; acetone residuals on the order of 100 ppm remained, even at the highest temperature.
- For the semivolatiles anthracene and BEHP, residuals remained well above 1000 mg/kg at the 150° and 350°F temperatures, but were reduced to less than 20 mg/kg at 550°F. Pentachlorophenol residuals remained high at the 150° and 350°F temperatures and were only reduced to levels on the order of 100 ppm at the 550°F temperature.

SARM II:

- As with SARM I, at 350° and 550°F, all volatiles except acetone were reduced to less than 1 mg/kg; acetone residuals on the order of 100 mg/kg remained, even at the 550°F temperature.
- All semivolatiles were reduced to less than 100 mg/kg at 350°F and to less than 10 mg/kg at 550°F.

Overall, the 150°F temperature was considered ineffective under the reaction conditions tested.

High-Temperature Incineration

In this segment of the test program, a series of pilot-scale test burns was conducted with SARM's I and II only. The testing was conducted at the John Zink testing facility in Tulsa, Oklahoma, in a rotary kiln incineration system using a nominal feed rate of 1000 lb/h. More than 12,000 pounds of each SARM soil was prepared for the tests so that three 4-hour test burn runs (for a total of six test burn runs) could be conducted on each SARM. Approximately 1 week prior to startup of the test burns, the soils were delivered to John Zink in forty-eight 55-gallon steel drums, each containing 500 to 600 lb of SARM I or SARM II.

Two runs per day were conducted over the 3-day period of September 16 through 18, 1987. Runs 1, 2, and 3 were conducted with SARM I (high organics, low metals), and Runs 4, 5, and 6 were conducted with SARM II (low organics, low metals). Equipment operations were normal throughout each run.

The process operating data collected during each test show that the temperatures and feed rates achieved were reasonably close to the goals (i.e., 1800°F in the kiln, 2000°F in the secondary combustion chambers, and a nominal feed rate goal of 1000 lb/h). Excess air was maintained at about 3 percent in the kiln and about 5 percent in the secondary chamber during both tests. Emissions of O_2, CO_2, and CO were steady throughout; and CO remained at less than 10 ppm at all times except for a few brief excursions of 45 to 90 ppm, which lasted from 1 to 5 minutes. A total of 13,932 lb of SARM I and 13,460 lb of SARM II were incinerated over a course of 3 days that involved 29 hours 22 minutes of testing.

Table X presents the results of chemical analyses (total waste analyses) of the bottom ash (i.e., SARM residue) samples collected during each test run. Samples analyzed for semivolatiles and metals were collected as composites over the course of each test; samples analyzed for volatiles were collected as discrete samples at the beginning, middle, and end of each run and composited at the time of analysis.

The volatile compounds styrene, tetrachloroethylene, and chlorobenzene, and the semivolatile compounds anthracene and pentachlorophenol were not detected in any of the ash samples. Measureable quantities of ethylbenzene and xylene were found in the ash of both SARM's. and 1,2-dichloroethane was found in the ash of SARM II, but the amounts were small (in the low parts-per-billion range) and typically at levels within 2 to 3 times the method detection limit. Acetone was found in the ash samples of all runs for both SARM's at significant levels ranging from 190 to 790 µg/kg; these levels are 24 to 99 times higher than the method detection level (8 µg/kg).

On the average, the concentrations of acetone and phthalate found in the ash of SARM I are similar to those found in the ash of SARM II, even though

Table X. Total Waste Analysis for SARM ash.

Parameter	Method detection limit	SARM I			SARM II		
		Run 1	Run 2	Run 3	Run 4	Run 5	Run 6
VOLATILES, µg/kg							
Ethylbenzene	7.0	ND[a]	19	ND	8	ND	13
Xylene	5.0	ND	34	ND	11	6	20
Tetrachloroethylene	4.0	ND	ND	ND	ND	ND	ND
Chlorobenzene	6.0	ND	ND	ND	ND	ND	ND
Acetone	8.0	440	420	630	190	210	790
1,2-Dichloroethane	3.0	ND	ND	ND	ND	5	10
Styrene	3.0	ND	ND	ND	ND	ND	ND
SEMIVOLATILES, µg/kg							
Anthracene	37	ND	ND	ND	ND	ND	ND
Bis(2-ethylhexyl) phthalate	63	1600	540	740	950	710	1300
Pentachlorophenol	370	ND	ND	ND	ND	ND	ND
METALS, mg/kg							
Lead	4.2	56	98	107	146	75	88
Zinc	0.12	217	227	250	252	199	237
Cadmium	0.12	<0.2	<0.2	<0.2	0.2	<0.2	<0.2
Arsenic	0.04	38	36	44	46	39	37
Copper	0.42	111	132	159	125	106	162
Nickel	0.30	12	15	11	12	9.1	12
Chromium	0.30	10	14	12	12	7	10
VOLATILE PICs, µg/kg							
2-Butanone	25	35	ND	ND	14[b]	ND	ND
Methylene chloride	2.8	2.9	5.4	4.2	ND	ND	ND
2-Chloroethylvinyl ether	5.0	70	ND	ND	ND	ND	ND

[a] ND = Not detected.

[b] Estimated value; less than method detection limit.

the input waste feed levels for these compounds were roughly 10 times higher in SARM I than in SARM II. This suggests sample contamination or carryover, and the data for these compounds should be interpreted with caution. Significant quantities of phthalate were also found in several of the method blanks, and phthalates are known to be commonly encountered contaminants in sample analysis.

The metals data for the ash samples were also interesting. Prior to the testing, most of the metals concentrations in the ash were expected to be elevated compared with those in the waste feed because of the combined effects of the retention of metals in the ash and the losses of water and organics from the feed during the incineration process. Cadmium levels in the ash, however, were expected to be low as a result of volatilization of the metal in the kiln at the high operating temperature of 1800°F. As expected, cadmium levels in the ash were quite low, at least 99.9 percent lower than the waste feed levels. Surprisingly, all of the other heavy metal levels were also lower in the ash (e.g., on the order of 50 to 80 percent lower) than in the waste feed, which indicates significant volatilization or perhaps slagging or condensation onto the kiln refractory. On the other hand, arsenic levels in the ash were more than double those in the feed across the board.

The test burns successfully met all the RCRA emission requirements for hazardous waste incineration. Stack samples collected during the SARM I and II test burns revealed the following:

- Particulate concentrations corrected to 7 percent O_2 were below the RCRA allowable limit of 0.08 gr/dscf for each SARM type.
- Measured HCl emission rates in pounds per hour were considerably less than the RCRA allowable rate of 4.0 lb/h for each SARM type.
- The average stack gas concentration of CO was less than 23 ppm during each test.
- The destruction and removal efficiency (DRE) performance standard of 99.99 percent was achieved for all of the volatile compounds for each SARM. The DRE data for the semivolatiles show that anthracene was effectively destroyed, as the amount in each emission was less than the method detection limit, and the resulting DRE's were greater than 99.99 percent. The DRE data for bis(2-ethylhexyl)phthalate showed that only three of six sample runs met the 99.99 percent criteria. Sample contamination (background level) problems may have been responsible for the poor DRE's in the other three runs.

SOLIDIFICATION/STABILIZATION

This project evaluated the performance of solidification as a means of treating the SARM soils. Tests were conducted on all four SARM's using three commonly used solidification agents or binders: portland cement (Type 1), lime kiln dust, and a 50:50 mixture by weight of lime and fly ash. At 7, 14,

21, and 28 days after the SARM's and binders were mixed, samples of the solidified material were subjected to unconfined compressibility strength (UCS) testing. Samples that achieved a UCS minimally greater than 50 psi or that showed the highest UCS below 50 psi after 14 and 28 days were subjected to total waste and TCLP analyses.

Results of the testing showed that the UCS tended to increase with time as the samples cured. Portland cement produced the strongest, hardest, and most consistent product, followed by kiln dust and lime/fly ash. The lime/fly ash samples required several weeks of curing before they finally set. The amount of moisture in the SARM's seemed to be an important factor in solidifying the soils. Offgassing of volatile compounds from the stabilized samples occurred during mixing and continued throughout the curing period.

Table XI presents an overall summary of the 28-day samples when analyzed for TCLP and TWA. The percentage reduction values in this table represent the total amount of contaminants found in the untreated soil samples (or TCLP extract) less the total amount of contaminant found in the stabilized sample (or TCLP extract) divided by the amount in the untreated soil (or TCLP extract) x 100 percent. The data have been adjusted to account for changes in soil volume and contaminant concentration caused by the addition of the binders.

The results fail to indicate either dramatic or consistent treatment efficiencies. Volatiles in the SARM's were reduced, but the reductions are attributed to volatilization losses (offgassing) during mixing and curing rather than binding within the stabilized matrix. Metals were less prevalent in the treated sample TWA and TCLP extracts, which indicates reduced mobility after stabilization/solidification. The percent reductions seldom exceeded 90 percent, however, and generally tended to range from 0 to 20 percent to 60 to 75 percent. Overall, kiln dust and lime/fly ash produced the best contaminant-reduction results.

SUMMARY AND CONCLUSIONS

The research program produced a valuable and interesting new data base outlining the kinds of results that can be achieved by treating a synthetic contaminated soil at bench and pilot scale. This paper only highlights key portions of the data base; it is by no means complete. Detailed reports covering the complete findings of each study are available through EPA's Hazardous Waste Engineering Research Laboratory in Cincinnati (see Acknowledgments).

Preparation of the SARM's is viewed as a particularly valuable segment of the research because this had never before been attempted on such a large (volumetric) scale. Methods of mixing both the basic clean soil and the contaminated material were developed and found to produce a quality product with good homogeneity. This allowed each of the treatment technologies to operate with a high degree of assurance that the starting materials were essentially identical from one test to another.

Table XI. Summary of Effectiveness of Stablization/Solidification Agents After 28-Day Cure (% Reduction)[a]

	SARM I			SARM II			SARM III			SARM IV		
	PC[b]	KD[c]	L/FA[d]	PC	KD	L/FA	PC	KD	L/FA	PC	KD	L/FA
	TCLP											
Volatiles	73.9	97.6	75.8	42.2	82.0	88.0	68.3	>93.6	90.3	-45	77.5	73.7
Semivolatiles	67.2	>98.8	>96.5	>53.8	>68.5	>72.8	-47.0	82.2	54.8	-139	57.2	88.6
Inorganics	>82.1	>75.0	>92.3	>83.0	>71.2	>92.1	99.4	83.7	54.3	85.3	66.4	68.3
	TWA											
Volatiles	-	98.5	83.7	86.9	99.7	97.0	76.9	98.1	92.0	58.5	95.3	83.4
Semivolatiles	-	87.7	80.2	33.1	38.0	27.5	-101	-37.5	-32.2	24.2	28.3	47.9
Inorganics	-	43.8	56.6	-13.6	9.7	28.1	28.5	73.2	82.3	32.3	60.5	73.9

[a] A negative percent reduction results when analysis of the treated residual (or extract of a treated residual) yields a higher contaminant concentration than the untreated material.

[b] PC = Portland cement.

[c] KD = Kiln dust.

[d] LFA = Lime/fly ash.

A rank-order summary of the effectiveness of each treatment technology on the four SARM's, is presented in Table XII. The thermal technologies effectively reduced the organic fractions (>99.6%) when measured by TWA. The chemical treatment (KPEG) operated on the semivolatile fraction with greater than 90 percent reduction effectiveness. Greater than 98 percent of the volatile organic compounds were removed, but this was likely due to volatilization during the test runs. Soil washing was the best metals reduction technique across all the SARM's, averaging 93 percent. Soils washing was also very effective in reducing the semivolatile compounds (averaging about 87%) and the volatiles (99%). Stabilization generally ranked behind the other technologies, as expected, since it does not remove metals, but immobilizes them. For stabilization, TCLP is a better measure of treatment effectiveness than TWA.

Phase II of the CERCLA Research Program was initiated in 1988 and is continuing. Soils from actual Superfund sites have been collected and are being tested for treatment effectiveness using the same bench-scale procedures as in Phase I. Results, which are expected to be available in late 1988, will be compared with those produced on the SARM's.

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Project	Contractor	EPA contract	EPA Project Officer
SARM preparation	PEI Associates, Inc. Cincinnati, Ohio	68-03-3413 Work Assignment 0-7	Richard P. Traver
Physical soil washing	PEI Associates, Inc. Cincinnati, Ohio	68-03-3413 Work Assignment 0-7	Richard P. Traver
Dechlorination/ KPEG	Wright State University Dayton, Ohio (subcontractor to PEI Associates)	68-03-3413 Work Assignment 0-6	Charles J. Rogers
Thermal desorption	IT Corporation Knoxville, Tennessee (subcontractor to PEI Associates)	68-03-3389 Work Assignment 0-5)	Robert C. Thurnau

Table XII. Overall BDAT Phase I treatment efficiency summary.[a]

SARM I (high organics, low metals)	Percent reduction	SARM II (low organics, low metals)	Percent reduction	SARM III (low organics, high metals)	Percent reduction	SARM IV (high organics, low metals)	Percent reduction
VOLATILES							
Incineration	>99.99	Incineration	>99.98	Soils washing + 2 mm water	>99.9	Chemical treatment KPEG No. 1	99.98
Soils washing + 2 mm water	>99.99	Soils washing - all fractions - water	>99.9	Soils washing + 2 mm chelate	99.9	Soils washing + 2 mm water	>99.9
Chemical treatment KPEG No. 1	99.96	Soils washing - all fractions - chelate	>99.7	Chemical treatment KPEG No. 1	99.5	Soils washing + 2 mm chelate	>99.9
Soils washing + 2 mm surfactant	99.82	Soils washing - all fractions - surfactant	99.7	Soils washing 2mm to 250 µm water	99.3	Soils washing + 2 mm surfactant	>99.9
Soils washing 2 mm to 250 µm surfactant	99.82	Solidification - kiln dust 28 days	99.7	Soils washing 2mm to 250 µm chelate	99.0	Soils washing 2 mm to 250 µm surfactant	>99.7
Soils washing 2 mm to 250 µm water	99.8	Low temperature thermal desorb at 150°F	98.70	Solidification - kiln dust - 28 days	98.1	Soils washing 2 mm to 250 µm chelate	>99.7
Low temperature thermal desorb at 350°F	99.79	Chemical treatment KPEG test No. 1	98.2	Chemical treatment KPEG No. 2	97.6	Soils washing 2 mm to 250 µm water	>99.7
Low temperature thermal desorb at 550°F	99.78	Solidification - lime/fly ash	97.0	Soils washing <250 µm chelate	98.2	Chemical treatment KPEG No. 2	98.1
Solidification - kiln dust - 28 days	98.5	Chemical treatment - KPEG No. 2	96.3	Solidification lime/fly ash - 28 days	92.0	Solidification kiln dust - 28 days	95.3
Chemical treatment KPEG No. 2	98.3	Low temperature thermal at 500°F	96.17	Soils washing <250 µm water	86.7	Soils washing <250 µm chelate	81.8
SEMIVOLATILES							
Incineration	>99.98	Incineration	>99.87	Chemical treatment KPEG No. 1	99.6	Soils washing + 2 mm surfactant	>98.3
Soils washing + 2 mm surfactant	>99.8	Soils washing + 2 mm water	93.9	Chemical treatment KPEG No. 2	99.0	Soils washing + 2 mm chelate	97.8
Soils washing + 2 mm water	>98.9	Soils washing + 2 mm surfactant	93.5	Soils washing + 2 mm chelate	>96.4	Chemical treatment KPEG No. 2	96.2
Chemical treatment KPEG No. 2	97.0	Soils washing + 2 mm chelate	90.1	Soils washing + 2 mm water	>94.8	Chemical Treatment KPEG No. 1	92.9

(continued)

Table XII (continued)

SARM I (high organics, low metals)	Percent reduction	SARM II (low organics, low metals)	Percent reduction	SARM III (low organics, high metals)	Percent reduction	SARM IV (high organics, low metals)	Percent reduction
Chemical treatment KPEG No. 1	95.6	Low temperature thermal desorb at 350°F	88.73			Solidification lime/fly ash - 28 days	47.9
Low temperature thermal desorb at 250°F	94.6	Chemical treatment KPEG Test No. 1	83.8			Soils washing 2 mm to 250 µm chelate	32.3
Soils washing 2 mm to 250 µm surfactant	82.3	Soils washing 2 mm to 250 µm surfactant	67.5			Soils washing 2 mm to 250 µm surfactant	39.4
Solidification lime/fly ash surfactant	80.2	Soils washing 2 mm to 250 µm water	22.7			Solidification kiln dust - 28 days	28.3
Solidication kiln dust	80.2	Soils washing 2 mm to 250 µm chelate	47.3				
Soils washing <250 µm water	59.7	Chemical treatment KPEG No. 2	42.3				
METALS							
Soils washing + 2 mm water	92.2	Soils washing + 2 mm water	>96.7	Soils washing 2 mm to 250 µm chelate	98.4	Soils washing + 2 mm surfactant	98.4
Soils washing + 2 mm surfactant	91.5	Soils washing + 2 mm chelate	95.9	Soils washing 2 mm to 250 µm chelate	98.4	Soils washing + 2 mm chelate	98.1
Soils washing 2 mm to 250 µm water	81.6	Soils washing + 2 mm surfactant	95.7	Soils washing 2 mm to 250 µm water	98.0	Soils washing + 2 mm water	97.1
Soils washing 2 mm to 250 µm surfactant	75.5	Soils washing 2 mm to 250 µm chelate	91.6	Soils washing 2 mm to 250 µm water	96.4	Soils washing 2 mm to 250 µm surfactant	91.8
Solidification lime/fly ash - 28 days	56.6	Soils washing 2 mm to 250 µm surfactant	85.1	Solidification lime/fly ash	82.3	Soils washing 2 mm to 250 µm water	90.7
Solidification kiln dust - 28 days	40.2	Soils washing 2 mm to 250 µm water	82.7	Snils washing <250 m chelate	78.2	Solidification lime/fly ash	73.9
Incineration	38.7	Incineration	64.3	Solidification kiln dust - 28 days	73.2	Solidification kiln dust	60.5
		Chemical treatment KPEG No. 1	39.4	Chemical treatment No. 1	49.4		

[a] Based on total waste analyses.

Project	Contractor	EPA contract	EPA Project Officer
Incineration	PEI Associates, Inc. Cincinnati, Ohio	68-03-3389 Work Assignment 0-7	Robert C. Thurnau
Stabilization	Acurex Corporation Durham, North Carolina	68-03-3241 Work Assignment 2-18	Edwin F. Barth

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